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A PROCESS FOR THE PREPARATION OF A HYDROGENATED POLYMER, A NEW HYDROGENATED
POLYMER AND THE USE THEREOF@

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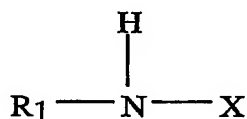
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A PROCESS FOR THE PREPARATION OF A HYDROGENATED POLYMER, A NEW
HYDROGENATED POLYMER AND THE USE THEREOF

The present invention relates to a process for the preparation of a hydrogenated polymer comprising the steps of hydrogenation of at least one carbon-carbon double bond of an unsaturated polymer in latex form in the presence of hydrazine, an oxidizing compound and a catalyst, followed by separation of the hydrogenated polymer from the latex. The invention also relates to the hydrogenated polymer. The invention further relates to the use of the hydrogenated polymer.

A process for the preparation of a hydrogenated polymer is known from WO 00/09568A. Disclosed is the hydrogenation of carbon-carbon double bonds of an unsaturated polymer in the presence of hydrazine, an oxidizing compound and a catalyst whereby an amine compound of formula A is added before, during or after hydrogenation to improve processability and reduce gelcontent.



formula A

R_1 is a hydrogen atom, an alkyl or cycloalkyl group with 1-30 carbon atoms, or an aromatic group with 6-30 carbon atoms, and X is chosen from the group comprising $-\text{R}_2$, $-\text{OR}_3$, $-\text{SR}_3$, $-\text{NR}_4\text{R}_5$, where R_2 , R_3 and R_4 are a hydrogen atom, an alkyl or cycloalkyl group with 1-30 carbon atoms or an aromatic group with 6-30 carbon atoms and R_5 is an alkyl or cycloalkyl group with 1-30 carbon atoms or an aromatic group with 6-30 carbon atoms and it being possible for R_3 - R_5 to contain one or more heteroatoms from the groups 13, 14, 15, 16 or 17 of the Periodic System of the Elements.

The above mentioned amine compound may be added for example after the hydrogenated polymer is separated from the latex, by mixing the amine compound and the hydrogenated polymer in an extruder. A disadvantage in this process is that residual amine compound, which is present in the hydrogenated polymer, has a negative influence on the cure properties which leads to inferior mechanical properties of the hydrogenated polymer.

The object of the present invention is to provide a process for the

hydrogenation of an unsaturated polymer with reduced residual amine content to overcome the above-cited disadvantage.

This object is achieved in that after the separation of the hydrogenated polymer from the latex a mixing step is carried out in which the hydrogenated polymer is first mixed with at least one amine compound whereafter the hydrogenated polymer is mixed with at least one compound of formula I



wherein R is an aliphatic or cycloaliphatic group with 1-20 carbon atoms or an aromatic group with 6-20 carbon atoms and X is chosen from an epoxide-, anhydride-, isocyanate-, acid chloride- or carboxylic acid group and $n=1-5$.

Surprisingly it has been found that if a compound of formula I is mixed with the hydrogenated polymer comprising the amine compound, hydrogenated polymers may be obtained with better mechanical properties. Another advantage is that the processing of the hydrogenated polymers is markedly improved.

The unsaturated polymer useful in the process of the present invention for example comprises from 5-100% by weight of a conjugated diene monomer unit and from 95-0% by weight of an ethylenically unsaturated monomer unit. Examples of conjugated diene monomer units are 1,3-butadiene, 2,3-dimethylbutadiene, isoprene and 1,3-pentadiene. Preferably 1,3-butadiene is used. Examples of the ethylenically unsaturated monomer units include unsaturated nitriles for example acrylonitrile and methacrylonitrile, monovinyl aromatic hydrocarbons for example styrene, (o-, m- and p-) alkylstyrenes, divinyl aromatic hydrocarbons for example divinylbenzene, dialkenyl aromatics for example diisopropenylbenzene, unsaturated carboxylic acids and the esters thereof for example acrylic acid, methacrylic acid, crotonic acid, itaconic acid, maleic acid, methyl acrylate, ethyl acrylate, butyl acrylate, 2-ethylhexyl acrylate and methyl methacrylate. Preferably as ethylenically unsaturated monomer, unsaturated nitriles are used, more preferably acrylonitrile is used.

Examples of suitable unsaturated polymers useful in the process of the present invention are polybutadiene, polyisoprene, styrene-butadiene copolymers (SBR), acrylonitrile-butadiene copolymers (NBR), natural rubber, butadiene-isoprene rubber and terpolymers of butadiene, acrylonitrile and butylacrylate or acrylic acid. Preferably, the unsaturated polymer, used in the process according to the present invention comprises from 50-80 % by weight of the conjugated diene-monomer unit and from 20-50 % by weight of the unsaturated nitrile group-containing monomer unit, more preferably, from 60-70 % by weight of the conjugated diene-monomer unit and

from 30-40 % by weight of the unsaturated nitrile group-containing monomer unit. Preferably an acrylonitrile-butadiene (NBR) copolymer is used in the process of the present invention.

5 The unsaturated polymer may be prepared by any method of preparation such as emulsion polymerisation, solution polymerisation or bulk polymerisation. Preferably, the unsaturated polymer has been prepared by means of emulsion polymerisation in water, so that the unsaturated polymer becomes available in latex form. US 5,442,009 gives clear instructions for the preparation of the unsaturated polymer in latex form for example the conditions to be used, the initiators
10 to be used, etc. If the unsaturated polymer is not available in the form of a latex, then the unsaturated polymer is preferably introduced into an aqueous dispersion, more preferably in a latex. The latex form of the unsaturated polymer may be hydrogenated as such. The polymer content of the latex can range from 1 to 70 wt.%, preferably from 5 and 40 wt.%.

15 The hydrogenation may be carried out batchwise as well as continuously. Closed or open vessels may be used.

The hydrazine used in the hydrogenation step of the present invention may be used as such, but it is also possible to use a hydrazine-releasing compound for example hydrazine hydrate, hydrazine acetate, hydrazine sulphate or
20 hydrazine hydrochloride. Preferably use is made of hydrazine or hydrazine hydrate. The hydrazine is for example present in a molar ratio between 0.1:1 to 100:1 relative to the carbon-carbon double bonds in the polymer chain. Preferably, this ratio lies between 0.8:1 and 5:1, most preferably between 0.9:1 and 2:1.

Examples of oxidizing agents which are suitable in the hydrogenation
25 step are air, oxygen, ozone, peroxides, hydrogen peroxide, iodine, iodates, hypochlorite and similar compounds. Particular suitable oxidizing agents are chosen from the group comprising peroxides and hydrogen peroxide. Most preferably, use is made of hydrogen peroxide. The oxidizing compound is for example present at a molar ratio of between 0.1:1 and 100:1 relative to the carbon-carbon double bonds in the
30 polymer chain. This ratio preferably is between 0.8:1 and 5:1, more preferably between 0.9:1 and 2:1.

The hydrogenation step according to the present invention is carried out in the presence of a catalyst. Suitable catalysts are compounds which contain an element from group 13, of the Periodic Table of the Elements as published on the
35 inside of the cover of the Handbook of Chemistry and Physics, 67th edition, 1986-1987 in accordance with the latest IUPAC nomenclature, as described in WO 00/09576 A.

Preferably, the catalyst is a compound which contains boron. More preferably the catalyst is chosen from the group comprising borates, peroxyborates and boric acid (H_3BO_3). Most preferably, boric acid is used.

Also possible as catalyst in the process of the present invention is a wide variety of metal ions or salts. For example antimony, arsenic, bismuth, cerium, chromium, cobalt, copper, gold, iron, lead, manganese, mercury, molybdenum, nickel, osmium, palladium, platinum, cerium, silver, tellurium, tin, vanadium and mixtures of any of them are representative metal ions which will react with hydrazine and which are useful as catalyst. Iron and copper are preferred metal ions with copper being most preferred. Those metal ions as catalyst are described in US-A-4452950. The man skilled in the art can easily determine how much of the above catalyst in molar ratio with respect to the hydrazine level, has to be used in the process of the present invention.

The sequence of addition of the hydrazine, oxidizing compound and the catalyst for the hydrogenation step can in principle be chosen freely. Preferably, however, the oxidizing compound is added to the reaction mixture after the hydrazine, in such a way that the concentration of the oxidizing compound remains low when the hydrogenation reaction takes place. Another preference may be that the hydrazine and hydrogen peroxide are added simultaneously to the latex during hydrogenation.

The hydrogenation reaction temperature may be between 0°C - 250°C ; preferably between 20°C - 150°C ; more preferably between 30°C - 80°C . The duration of the hydrogenation varies for example from 1 hour to 24 hours. The hydrogenation time is preferably from 1-12 hour(s).

After the hydrogenation step the hydrogenated polymer is separated from the water phase of the latex. Examples of suitable separation processes are precipitation, latex dewatering via extrusion and steam stripping. Particularly precipitation is a suitable purification method, for which standard processes can be used, for instance the addition of an acid or salt or a mixture of both to the aqueous dispersion of the hydrogenated polymer, or administration of an alcohol. Preferably, use is made of an alcohol or a magnesium sulphate-, a calcium chloride- or an aluminium sulphate solution in water for precipitation of the hydrogenated polymer latex.

After separation of the hydrogenated polymer a mixture is obtained of hydrogenated polymer crumb and water, which may contain all kinds of compounds for example hydrazine, peroxide, etc. The hydrogenated polymer may be dried and the so obtained hydrogenated polymer crumb may be further processed.

In the process of the present invention the hydrogenated polymer crumb is further processed by a mixing step which is carried out after the separation of the hydrogenated polymer from the latex. The hydrogenated polymer is first mixed with at least one amine compound whereafter the hydrogenated polymer is mixed with at least one compound of formula I



wherein R is an aliphatic or cycloaliphatic group with 1-20 carbon atoms or an aromatic group with 6-20 carbon atoms and X is chosen from an epoxide-, anhydride-, isocyanate-, acid chloride- or carboxylic acid group and $n=1-5$. Preferably $n=1-3$, more preferably $n=1-2$.

It has been found that if at least one compound of formula I is mixed with the hydrogenated polymer comprising at least one amine compound, hydrogenated polymers may be obtained with better mechanical properties for example better stress relaxation properties and better compression set values. Moreover the processing of the hydrogenated polymer is markedly improved.

Examples of the amine compounds useful in the process of the present invention are compounds of formula NH_2-R-X wherein R is chosen from an aliphatic group comprising at least one C atom or an aromatic group comprising at least 6 C-atoms and X is chosen from a hydrogen atom, NH_2 -, OH- or SH-group.

Examples of compounds of formula NH_2-R-X are methylamine, ethylamine, ethylene diamine, dodecylamine, methanolamine, ethanolamine, cyclohexyldiamine, o-phenylene diamine, 3,4-toluene diamine, 1,8-naphthalene diamine, aniline, o-aminophenol and ortho-aminothiobenzene. Preferably diamines are used as amine compound. More preferably ortho-aromatic diamines are used as amine compound.

The amount of amine compound for example varies between 0,01-5 wt%. Preferably it varies between 0,1-2 wt%.

Examples of compounds of formula I useful in the process of the present invention are phthalic acid anhydride, maleic anhydride, acetic anhydride, stearic anhydride, succinic anhydride, epoxidised natural oils for example epoxidised linseed oil, propylene oxide, cyclohexane di-epoxide, stearylchloride, acetyl chloride, toluene diisocyanate, hexane di-isocyanate, phenylene-isocyanate or stearic acid. Preferably phthalic acid anhydride, maleic anhydride, acetic anhydride, stearic anhydride, succinic anhydride are used in the process of the present invention. More preferably phthalic acid anhydride is used.

The mixing step in the process of the present invention may for example take place in a batchkneader or a continuous kneader for example a single,

double or multi-screw extruder. Preferably the mixing step is carried out in a double screw extruder. The double screw extruder for example consists out of a series of barrels which can for example be divided in three zones after the hydrogenated polymer is fed to the extruder. A first zone or melting zone wherein the hydrogenated polymer is melted. A second zone which is a mixing and reaction zone in which the amine compound may be injected for example as a melt or solution to react with the hydrogenated polymer. A third zone which is a mixing and reaction zone where the compound of formula I may be injected for example as a melt or solution into the hydrogenated polymer. After the third zone vacuum may be applied to remove all or parts of the injected compounds and/or solvents.

The compound of formula I is added after the mixing of the hydrogenated polymer and the amine compound. Preferably the compound of formula I is added after at least 10 % of the amine compound has reacted with the hydrogenated polymer. Even more preferably after at least 50 % of the amine compound has reacted with the hydrogenated polymer. Most preferably after more than 90 % of the amine compound has been reacted with the hydrogenated polymer. The amine content can be determined by a man skilled in the art using known methods like for example gas chromatography.

The temperature of the hydrogenated polymer melt in the extruder may vary between wide ranges for example between 100-380°C. Preferably between 250-350°C.

It is preferred that oxygen is excluded as much as possible during the mixing step and after the hydrogenated polymer has left the extruder still being hot. To prevent exposure to oxygen cooling under nitrogen or water may be applied. During the mixing step a mastication promotor for example pentachlorothiophenol or its zinc salt or dibenzamidediphenyl disulfide may be added to the hydrogenated polymer. Preferably the mastication promotor is added together with the amine compound in the second zone.

The present invention also relates to the hydrogenated polymers obtainable by the process of the present invention. It has been found that the hydrogenated polymers of the present invention show better mechanical properties after curing than the prior art hydrogenated polymers. For example the stress relaxation properties and the compression set values are markedly improved. The curing is carried out with a curing agent. Examples of curing agents are sulfur, sulfurous compounds, metal oxides, maleimides, phenol resins or peroxides. Examples

of sulphur cure agents are thiazole-, mercapto- or sulfenamide compounds or dithiocarbamates with metal oxides for example zinc oxide. Examples of suitable peroxide curing agents are organic peroxides for example dicumyl peroxide, di-tert-butylperoxide, 2,5-dimethyl-(2,5-di-tert-butylperoxy)hexane, 1,3 -bis(tert-butylperoxyisopropyl)benzene, 1,1-bis(tert-butylperoxy)-2,3,5-trimethylcyclohexane, benzoyl peroxide, 2,4-dichlorobenzoyl peroxide, tert-butyl peroxybenzoate, tert-butyl peroxyisopropylcarbonate, diacetyl peroxide, lauroyl peroxide, tert-butyl cumyl peroxide or a mixture of any of them. Preferably a peroxide curing agent is used.

Another advantage is that the hydrogenated polymers obtainable by the process of the present invention show a better processability, which is shown in a better die swell.

The hydrogenated polymers according to the present invention may contain additives, such as for instance stabilizers, chosen from the group comprising free radical stabilizers, antioxidants, dihydroquinoline derivatives, benzimidazole derivatives and naphthylamine derivatives. Examples of the above additives are described in the Rubber Technology Handbook, author Werner Hofmann, Carl Hanser Verlag, Munich, Vienna, New York, 1989. The hydrogenated polymer may for instance also contain reinforcing agents. Examples of reinforcing agents are carbon black and silica.

The additives may be added before, during or after the hydrogenation. The additives may for example be added to the unsaturated polymer prior to the start of the hydrogenation but may also be added prior to the separation of the hydrogenated polymer. It is also possible to add the additives during the mixing step.

The present invention also relates to the use of the hydrogenated polymer obtainable by the process of the present invention. The hydrogenated polymer may for example be used to form articles. The articles may for example be formed by using processing methods customarily employed by one skilled in the art, examples being extrusion, co-extrusion, injection molding or robotic extrusion. Examples of articles, in which the hydrogenated polymer according to the present invention shows up very well are automotive parts to be mounted in the engine compartment for example belts, hoses and seals.

The process according to the present invention will be elaborated in the following examples, without however being restricted thereto.

- The processing properties were determined by the Garvey Die B test according to ASTM D 2230-73 with a die area 0,50 cm², a screw temperature of 80 °C, a zone temperature of 120 °C and screw speeds of 30rpm and 50rpm.
- The compression set was determined according to ISO 815 Type B.
- 5 • The stress relaxation was determined according to ISO 3384 Method A.

Experiment A

The hydrogenation was performed in a 2000 liter reactor equipped with stirrer, nitrogen outlet and cooling jacket. Boric acid (9,3 kg), water (~10 kg) and
10 hydrazine 85% hydrate (72,11 kg) were charged into the reactor. After the boric acid was dissolved 380 kg of NBR latex containing 25 wt% solids was charged. The NBR had a bound acrylonitrile of 33wt% and a Mooney of 30 (ML 1+4). Hydrogenation was started under stirring with the addition of hydrogen peroxide (35wt% in water) and lasted for 12 hrs. Hydrogen peroxide addition stopped when
15 residual hydrazine levels were <5ppm. During the hydrogenation the temperature was kept on 45 °C .

The HNBR latex was coagulated in a stirred coagulation vessel by addition of a concentrated calcium chloride solution and the wet HNBR rubber was dried in an oven at 60-70 °C.

20 The dried HNBR was mixed with 2 wt% 3,4-toluene diamine (TDA) on a 3 liter Farrell batch kneader with a maximum temperature of 90 °C. The resulting HNBR-TDA mixture was fed into a ZSK 30 extruder L/D 39 consisting out of 14 barrels. Temperature set over the extruder was 275 °C, screw speed was 250 rpm and throughput was 6 kg/h. The extrudate was cooled in a water-bath.

25

Example I

Experiment A was repeated, the difference being that, the extruded HNBR was mixed with 2 wt% phthalic anhydride on a 3 liter Farrel kneader.

30 From the HNBR retrieved in Experiment A and example I masterbatches were prepared (see compound recipe below) using an intermeshing Shaw (K1 Intermix Mark IV) laboratory mixer with an empty volume of 5,3 L (batch weight about 3 kg). Garvey Die was performed to measure the processing properties of the masterbatches. Results are given in table 1.

Table 1

Example	A		I	
screw speed rpm	30	50	30	50
Head pressure bar	53	61	50	56
Power kW	1,3	2,3	1,2	2,2
Die swell %	98	115	82	60
Output g/min	340	599	359	619
Appearance				
Swell and porosity	3	2	3	3
Edge	1	2	3	3

(4=very good; 3=good; 2=bad; 1=very bad)

As can be seen from the above results the addition of the phthalic anhydride improves the die swell (less die swell) and the appearance of the hydrogenated polymer.

- 5 Mechanical properties were measured on cured samples. Cure systems were mixed with the masterbatches using a Troester WNU-5 two-roll mill (see compound recipe's below). The friction ratio was set at 1:1,2 and the rolls of the mill were cooled. The compounds were stored for at least one day at room temperature before use. Sulphur cure was done for 20 minutes at 16 °C. Peroxide cure was done
- 10 for 20 minutes at 170 °C . Results are given in tables 2 and 3.

<u>Compound recipe's</u>		
<u>Ingredient</u>	<u>sulphur cure</u>	<u>Peroxide cure</u>
HNBR	100	100
ZnO active	5	5
Stearic acid	1	1
Carbon black N-772	40	40
SDPA(N 445)	1	1
ZMMBI	1	
TOTM	7	7
MBT-80	0,63	
TMTD-80	1,88	
S80 (R.S)	0,63	
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SDPA(N 445)=styrenated diphenyl amine

ZMMBI=methyl-zinc-2-mercaptobenzimidazole

MBT-80=2-mercaptobenzothiazole

TMTD-80=tetramethyl thiuram disulfide

Perkadox 14-40™

Table 2: Compression set and stress relaxation properties of sulphur cured samples

Example/ experiment	A	I
Sulphur Cure		
Compression set %		
22h/70 °C	40	32
22h/100 °C	53	48
22h/150 °C	72	67
Stress Relaxation %		
24 h 100 °C	65	77
72 h	53	70
168 h	49	64
336 h	42	54
672 h	41	49
2016h	24	40

As can be seen from table 2 the stress relaxation properties as well as the compression set properties are improved by the use of phthalic anhydride.

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Table 3: Compression set properties of peroxide cured samples

Example/ experiment	A	I
Peroxide Cure		
Mh-MI	0,3	0,9
Hardness IRHD	51	59
Tensile strength Mpa	11	20
Compression set %		
22h/-25 °C	99	95
70h/23 °C	62	38
22h/70° C	79	39
22h/100 °C	80	41
22h/150 °C	82	50

As can be seen from the above results the compression set was improved markedly by the use of phthalic anhydride.

10

CLAIMS

1. Process for the preparation of a hydrogenated polymer comprising the steps of:
 - 5 a. hydrogenation of at least one carbon-carbon double bond of an unsaturated polymer in latex form in the presence of hydrazine, an oxidizing compound and a catalyst, followed by
 - b. separation of the hydrogenated polymer from the latex characterised in that after the separation
 - 10 c. a mixing step is carried out in which the hydrogenated polymer is first mixed with at least one amine compound whereafter the hydrogenated polymer is mixed with at least one compound of formula I
$$\text{R-X}_n \quad \text{formula I}$$
wherein R is an aliphatic or cycloaliphatic group with 1-20 carbon atoms or an aromatic group with 6-20 carbon atoms and X is chosen from an epoxide-, anhydride-, isocyanate-, acid chloride- or carboxylic acid group and $n=1-5$.
- 15 2. Process according to claim 1 characterised in that the compound of formula I is added after at least 10 % of the amine compound has been reacted with the hydrogenated polymer.
- 20 3. Process according to anyone of claims 1-2 characterized in that the mixing step is carried out in a twin-screw extruder.
4. Process according to anyone of the claims 1-2 characterised in that the compound of formula I is chosen from phthalic acid anhydride, maleic anhydride, acetic anhydride, stearic anhydride or succinic anhydride or mixtures of any of them.
- 25 5. Process according to anyone of the claims 1-4 characterised in that the compound of formula I is added in an amount which varies between 0,1-2 wt%
6. Process according to anyone of the claims 1-5 characterised in that the amine compound is a compound of formula $\text{NH}_2\text{-R-X}$ wherein R is chosen from an aliphatic group comprising at least 1 C atom or an aromatic group comprising at least 6 C-atoms and X is chosen from a hydrogen atom, NH_2 -, OH- or SH-group.
- 30 7. Process according to anyone of the claims 1-6 characterised in that the amine compound is an ortho-aromatic diamine.
- 35

8. Process according to anyone of the claims 1-7 characterised in that the unsaturated polymer is acrylonitrile-butadiene rubber (NBR).
9. Process according to anyone of claims 1-8 characterised in that the catalyst is a compound which contains boron.
- 5 10. Process according to claim 9 characterised in that the catalyst is boric acid.
11. Process according to claims 1-8 characterised in that the catalyst is a metal ion catalyst.
12. Hydrogenated polymer obtainable by the process according to anyone of the claims 1-11.
- 10 13. Articles comprising the hydrogenated polymer according to claim 12.
14. Use of the hydrogenated polymer according to claim 12.

ABSTRACT

This invention relates to a process for the preparation of a hydrogenated polymer comprising the steps of hydrogenation of carbon-carbon double bonds of an unsaturated polymer in latex form in the presence of hydrazine, an oxidizing compound and a catalyst, followed by separation of the hydrogenated polymer from the latex. After the separation a mixing step is carried out in which the hydrogenated polymer is first mixed with at least one amine compound whereafter the hydrogenated polymer is mixed with at least one compound of formula I

10 R-X_n formula I

wherein R is an aliphatic or cycloaliphatic group with 1-20 carbon atoms or an aromatic group with 6-20 carbon atoms and X is chosen from an epoxide-, anhydride-, isocyanate-, acid chloride- or carboxylic acid group and n=1-5.

15